DELAMINATION OF LAYERED MATERIALS UNDER IMPACT LOADING

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Prof. Dr. Canan ÖZGEN
Director

I certify that this thesis satisfies all the requirements as a thesis for the degree of Master of Science.

Prof. Dr. Bilgehan Ögel
Head of Department

We certify that we have read this thesis and in our opinion it is fully adequate, in scope and quality, as a thesis for the degree of Master of Science in Metallurgical and Materials Engineering.

Assoc. Prof. Dr. Ali KALKANLI
Supervisor

Examining Committee Members:

Assoc. Prof. Dr. Ali KALKANLI

Prof. Dr. Ekrem SELÇUK

Prof. Dr. Haluk ATALA

Prof . Dr. Rıza GÜRBÜZ

Assoc. Prof. Dr. Tamer ÖZDEMİR

ABSTRACT

DELAMINATION OF LAYERED MATERIALS UNDER IMPACT LOADING

DİNÇ, Dinçer
M.S., Metallurgical and Materials Engineering Department
Supervisor: Assoc. Prof. Dr. Ali KALKANLI

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In this study, a cold worked tool steel and a low carbon steel (St 37), which were joined by brazing, were subjected to impact and shear loading. The end product is used as paper cutting blades in the industry. Effects of different brazing filler metals on the delamination of the blades under impact loading and on the impact toughness of the blades were studied. The target is to achieve higher impact toughness values without delamination.

Impact toughness of the steels, joined by Cu, CuNi and BNi brazing filler metals and separation of brazed surfaces under shear loading were studied. The microstructures that were formed as a result of each application were studied by scanning electron microscopy and x-ray diffraction.

The results indicate that brittle intermetallic compounds are formed in BNi brazing filler metal application. It is observed that CuNi alloy with 24% wt Ni form stronger bonds with the base metals than pure Cu and 10% wt Ni CuNi alloy.

Keywords: Brazing, Delamination, Impact Loading, Cu, BNi, CuNi

ÖZ

KATMANLANMIŞ MALZEMELERİN ÇARPMA YÜKÜ ALTINDA AYRILMASI

DİNÇ, Dinçer
Yüksek Lisans, Metalurji ve Malzeme Mühendisliği Bölümü
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Bu çalışmada, sert lehimleme ile birleştirilen bir soguk işlenmiş takım çeliği ile bir düşük karbon çeliği (St 37) çarpma ve basma yüküne maruz bırakıldı. Son ürün sanayide kağıt kesme bıçağı olarak kullanılmaktadır. Farklı sert lehimleme dolgu metallerinin, bıçakların çarpma yükü altındaki ayrılmaları ve bıçakların çarpma toklukları üzerindeki etkileri incelendi. Amaç ayrılma gerçekleşmeksizin daha yüksek çarpma tokluklarına ulaşmaktır.

Cu, CuNi, ve BNi sert lehimleme dolgu metalleri ile birleştirilen çeliklerin çarpma toklukları ve birleştirilen yüzeylerin basma yükü altında ayrılmaları incelendi. Her uygulama sonucunda oluşan mikroyapılar taramalı elektron mikroskobu, optik mikroskop ve x-ışınları ile incelendi.

Sonuçlar BNi sert lehimleme dolgu metali uygulamasında kırılgan arametalik bileşiklerin oluştuğunu gösterdi.. 24% ağırlıkça Ni içeren CuNi alaşımının ana metallerle saf Cu' dan ve 10% ağırlıkça Ni içeren CuNi alaşımından daha kuvvetli bağ oluşturduğu gözlendi.

Anahtar Kelimeler : Sert Lehimleme, Ayrılma, Çarpma Yükü, Cu, BNi, CuNi

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TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

CHAPTER 1

INTRODUCTION

Tool steels are used in the fabrication of cutting tools and dies where high hardness and resistance to wear are primary requisites. Frequently, the cutting or wearing surface may be only a small section of the tool assembly and it is necessary to join the tool steel to the assembly. Brazing is one commonly used method for doing this. For better performence of the cutting tools, appropriate brazing filler metals should be used in joining the tool steels to the assembly. In this study the tool considered is paper cutting blades (Fig. 1.1) and the most severe problem faced during the operation of these blades is the delamination of tool steel and the assembly at the joint . This study aims at improving the impact toughness of cutting blades by using different brazing filler metals and by changing the brazing parameters. The issues of concern are the wetting of brazing filler metals between the steels, dissolution of the base metals by the braze alloy, diffusion of elements in the brazing filler metal and the formation of intermetallic phases in the joint area.

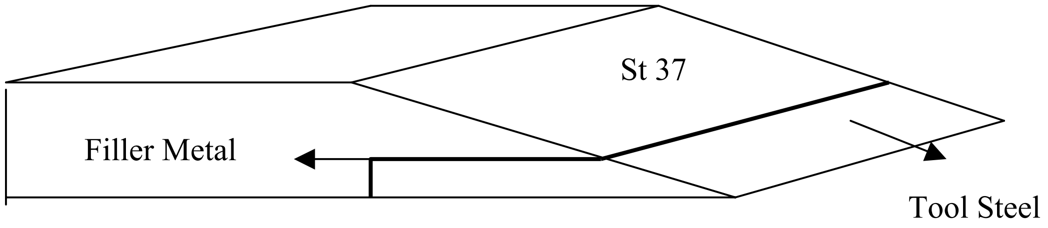

Fig 1.1 Schematic Representation of a Cutting Blade

In this chapter an introductory overwiev of brazing processes will be presented.

Details of brazing with Cu, BNi and CuNi brazing filler metals will be presented in chapters 2, 3 and 4 respectively.

1.1 Brazing

American Welding Society (AWS) defines brazing as " A group of welding processes wherein coalescence is produced by heating to a suitable temperatures above 450 ° C and by using a ferrous or nonferrous filler metal, having a melting point below that of the base metal and a liquidus temperature above 450 ° C. The filler metal is distrubuted between the closely fitted surfaces of the joint by capillary attraction".

In paper cutting blades, the base metals are 8 % Cr cold worked tool steel and St 37 low carbon steel. As the austenitization and brazing processes of cutting blades are performed at the same furnace, the above definition of AWS on brazing is more restricted for cutting blade manufacturing. The appropriate brazing filler metals in cutting blade manufacturing should have melting temperatures not much higher than the austenitizing temperature of the tool steel, which is 1040 ° C.

The function of a brazing filler metal is to form a molecular union between two other metals. The characterisctics of the joint metal will differ from those of the metals joined but the compatibility should be the maximum obtainable. Compatibility means that there must be a sufficiently close affinity between the brazing filler metal and both the metals of the joint to permit wetting at the brazing temperature and that the bond will posses some similarity of physical and mechanical properties [1].

A solid is accepted as having been wetted by a liquid if a film of it refuses to drain away when a force such as gravity causes the bulk of the liquid to flow off the solid surface [1]. Wetting occurs where there is a mutual attraction between the molecules of the liquid and the solid. This attraction is not necessarily due to chemical bonds, but there must be some chemical affinity. Clean solid metals are tought to have free affinities at their surfaces, but on exposure to the atmosphere, these affinities are usually quickly satisfied by gas molecules, grease and dirt. One of the functions of a brazing flux is to clean the metal, thus release these affinities so that they can be occupied by the molecules of the molten filler metal.

Base metal and filler metal interactions are mostly important in determining the behavior of brazed joints. When a brazing filler metal wets the base metal on which it is applied, interactions between the filler metal and base metal occur. The extent of interaction varies greatly depending on compositions and thermal cycles. First, the molten filler metal can dissolve base metals. Second, constituents of the filler metal can diffuse into the base metal, either through the bulk of the grains or along the grain boundaries as a liquid. The results of such base metal dissolution or filler metal diffusion may be to raise or lower the liquidus or solidus temperatures of the filler metal layer, depending upon composition and thermal cycle.

Intermetallic compounds formation is also frequently seen as a result of interaction between constituents of the base and filler metals. These compounds are usually brittle. Whether or not the compounds form, depends on base and filler metal compositions, the brazing time and temperature. Just because intermetallic compounds do form, it does not necessarily follow that the joint is so embrittled as to lose engineering utility. This depends on the nature of specific compound and its quantity and distribution.

1.2 Brazing Process

The ability to produce a properly brazed joint with adequate strength is dependent on many factors like a suitable brazing filler metal, joint clearance, brazing temperature, furnace atmosphere, the nature and quality of the base metal surfaces, thermal expansion coefficients of the base and filler metals.

For satisfactory use as a brazing filler metal, a metal or alloy should have the following characteristics [10]:

1- Ability to wet and make a strong, sound bond on the base metal.
2- Suitable melting temperature and flow properties to permit distribution

by capillary attraction in properly prepared joints.

3- A composition of sufficient homogenity and stability to minimize seperation by liquation under the brazing conditions.
4- Be capable of producing a braze joint which will meet service requirements such as strength and corrosion resistance.
5- Depending on the requirements, to be able to produce or avoid base metal-filler metal interactions.

The right joint clearance is that which will fill completely with brazing material and retain it there by capillary attraction. Alloying between the filler metal and base metal occurs when the equilibrium systems of the paired solid and liquid metals show definite evidence of intersolubility either as solid solution phases or as intermetallic compounds. Apart from possible effects on the strength of the eventual joint, this intersolubility has a bearing on optimum clearances because the viscosity of the brazing filler metal is usually increased when it occurs, with the result that the rate of flow into the joint decreases as the intersolution proceeds so if appreciable alloying occurs the joint clearance has to be increased. For maximum joint strength and capillary flow the joint clearance between the parallel mating surfaces should be held between 25 - 75 µm and should be kept as small as possible at brazing temperature [6].

Generally, a brazing filler metal is completely molten before it flows into a joint and is distributed by capillary attraction. Therefore, normally, the liquidus may be considered the lowest temperature which should be used for brazing, and all sections of the joint must be heated to this temperature or higher. However, there are a few alloys used as brazing filler metals which become sufficiently fluid below the actual liquidus. Usually the lowest brazing temperatures are preferred to economize on the heat energy required, to minimize heat affect on base metal (annealing, grain growth or warpage), to minimize base metal – filler metal interactions and to increase the life of fixtures, jigs or other tools. Higher brazing temperatures may be desirable to use a higher, but more economical brazing filler metal, to combine annealing, stres relief or heat treatment of the base metal with brazing, to permit subsequent processing at elevated temperatures, to promote base metal – filler metal interactions, to effectively remove surface contaminants and oxides with vacuum or atmosphere brazing and to avoid stress cracking [4].

For high production brazing, furnace brazing process is applicable. There are two distinct types of gaseous atmospheres in use for brazing applications [1]:

1- Chemically inert gas atmospheres such as argon or helium, protect the parts being brazed from coming into contact with other gaseous elements which might react with the metals being joined to produce surface films, which might in turn inhibit flowing and wetting by the molten brazing alloy.
2- Chemically active and reducing atmospheres such as hydrogen and either exothermic or endothermic combusted gases will react with any surface film present on either the parts to be joined or the brazing-alloy preform, during the brazing cycle, removing them in the process.

Surface roughness, cleanliness and the presence of elements such as nitrogen, aluminum, titanium etc. at the surface are extremely important because of their effect on brazing filler metal flow and wettability. The surface should be free from oil, grease, dirt and other undesirable contaminants. If the surfaces are oxidized, the oxide layers should be removed by a mechanical or a chemical cleaning process. Brazing filler metals flow by capillary action and this capillary flow can be strongly influenced by the base metal surface finish in braze joints. Neither very smooth nor very rough surfaces are good for the joint. In surfaces with a very rough profile, only the peak points are in contact during brazing, thus causing weak joints [4]. Titanium, chromium, aluminum and zirconium have the potential to form oxide films on base metal surfaces if present as an alloying element. The formation of these oxide films occurs between 535 – 927 ° C. While the formation of these oxides can not be completely eliminated, they can be minimized depending on the quality of the furnace atmosphere [1]. Vacuum atmospheres will oxidise these oxide forming elements less than pure dry hydrogen atmospheres, and faster heating rates will tend to cause less discoloration. Electroplating with nickel is also a very effective method of brazing base metals containing oxidation sensitive elements. The nickel plating acts as a barrier between the filler metal and the oxide forming elements, thus enabling a flow of liquid metal into the joint [1].

One of the most important criteria when brazing dissimilar metals is considering the difference in thermal expansion between them. If a metal which has a high thermal expansion surrounds a low thermal expansion metal, clearances which are satisfactory for promotion of capillary flow at room temperature will be too great at brazing temperature. Conversely if the low expansion metal surrounds the high expansion metal, no clearance may exist at brazing temperature. With dissimilar materials it is necessary that the one having the greater coefficient of thermal expansion is used as the female member of the joint [4]. Change in length % for a temperature change of 700 ° C Iron : 0.8, Mild Steel : 0.8, Nickel : 1.0, Copper : 1.1, 80/20 copper-nickel : 1.1, 80/20 Nickel-chromium : 1.2 . Zhang, [18] suggests using ductile filler metal in order to limit the stresses arising between two materials with different thermal expansion coefficients.

1.3 Metallurgical Phenomena of Base and Filler Metals

1.3.1 Carbide Precipitation : In some steels and other alloys which contain chromium and carbon, carbon combines with the chromium and is rejected as chromium carbide usually at the grain boundaries. As chromium carbides are brittle, angular, hollow crystals, they raise the braze joint stress. Because of that, carbon is an undesirable element in most brazing filler metals. If the brazing can be done very rapidly, no appreciable amount of carbides will be precipitated.

1.3.2 Oxide Stability : Most of the residual oxides present on base and filler metals are easily removed by proper flux or reducing atmosphere. Chromium oxide may be removed with some fluoride-bearing fluxes, but can not be reduced by hydrogen unless the atmosphere is very dry (about –70 ° F dew point) and only at high temperatures (in the range of 1800 – 2000 ° F) [1].

1.3.3 Hydrogen Embrittlement : When hydrogen diffuses into a metal which has not been completely deoxidized it may reduce the oxide of the metal if the temperature is high enough. Metallic sponge and water vapor are the end products of this reaction. As the molecular size of water vapor is too large to permit diffusion to the surface, a pressure is developed. These high pressures tear the metal apart at the grain boundaries. Metal which has been hydrogen embrittled exhibits lowered tensile properties. Electrolytic tough pitch copper, silver and palladium, when they contain oxygen, are subject to hydrogen embrittlement if heated in the presence of hydrogen. It is better practice to use deoxidized copper or oxygen free copper where brazing is to be performed.

1.3.4 Sulfur Embrittlement : Nickel and certain alloys containing appreciable amounts of nickel, if heated in the presence of sulfur or compounds containing sulfur, may become embrittled. A low melting nickel sulfide is formed preferentially at the grain boundaries and will crack if stressed. Chromium containing alloys are less susceptible to sulfur embrittlement. Sulfur containing materials such as oil, grease etc should be removed prior to brazing.

1.3.5 Phosphorus Embrittlement : Phosphorus combines with many metals to form brittle compounds known as phosphides. For this reason copper-phosphorus filler metals are not usually used with any iron or nickel base alloys.

CHAPTER 2

COPPER BASE BRAZING FILLER METALS

Pure deoxidized copper, low in such impurities as bismuth and lead (0.02% Bi, 0.005% Pb) and free of volatile and harmful impurities is very extensively used in brazing carbon and alloyed steel, nickel and its alloys.

Copper wets readily and flows freely on steel, is stronger than low melting point alloys, highly ductile and not scarce. Copper oxides are easy to reduce in hydrogen containing atmospheres and dissociate in argon. The limitations of copper as a filler metal are, its high melting point (1083 ° C) and the tendency to cracking on solidification in the case of brazing in a reducing atmosphere due to the formation of $Cu-Cu_2O$ eutectic[2]. On the other hand, Cu_2O decreases the wetting angle so increases wetting ability of the alloy.

Copper brazing is carried out at temperatures ranging from 1100 ° C to 1200 ° C depending on the alloy additions to the steel and alloys. Copper brazing is normally done in a reducing or shielding atmosphere, or in the presence of flux.

The interaction between copper and other elements make it possible to design copper-base filler metals with brazing temperatures variable over a wide range (700 ° C to 1200 ° C). Copper has the capability of combining with phosphorus and silver to form low melting point eutectics, the property of taking zinc into partial solid solutions with a narrow solidification range, the ability to take manganese, gold, palladium, nickel into a continuous range of solid solutions. The melting point of copper is lowered to 870 ° C or 889 ° C by alloying with manganese (35%) or gold (80%) respectively. Apart from the principal elements of copper filler metals, tin is added to them to depress the melting point and increase the flowability, silicon is added for strength and to lower the melting point and reduce the vaporability of zinc. Iron and cobalt are added for strength. But the alloying with iron should be limited due to the formation of an independent phase detrimental to corrosion resistance [2]. Some copper filler metals are capable of self fluxing when alloyed with phosphorus, lithium and boron. Boron also widens to some extent the solidification range of copper brazing alloys. Iron and silicon can be substituted for boron as hardeners added to copper filler metals used to braze carbide tools. As in the case of boron, the stated elements form finely dispersed inclusions of chemical compounds strengthening the tough matrix of the filler metal [2].

Copper phosphorus filler metals are very fluid and flow readily into gaps. Brazing should be fast as these alloys have a greater tendency to segregate when heated slowly. A major disadvantage of CuP filler metals is their low ductility. Due to the possible formation of brittle phosphides, steel and nickel alloys are not brazed with CuP filler metals.

Filler metals based on CuZn alloys melt at higher temperatures than CuP based brazing alloys. Because of their relatively low melting temperatures, narrow solidification range, natural abundance and the high solubility of zinc in copper, brass filler metals are widely used in the brazing of copper alloys and steel. Steel is brazed with brass filler metals at temperatures from 850 ° C to 950 ° C. The main disadvantage of brass filler metals is that zinc vaporizes partially during brazing. Pure zinc boils at 906 ° C, and when in brass the zinc boiling point rises to 1000 ° C with 50% Cu. The evaporation of zinc from brass can be reduced substantially by alloying with 0.1 to 0.5 % Si. The silicon inhibits the diffusion of zinc and decreases the solubility of hydrogen in the brass, thus reducing the risk of porosity upon solidification of the brazed joint [6]. Silicon has a greater chemical affinity for iron than for copper. The brazing of carbon steel with silicon bearing brass filler metals forms interlayers of steel base solid solutions enriched with silicon and Fe_3Si

intermetallics. This leads to the embritlment and reduced strength of the joint.

The presence of solid solution having the lowest melting point of 870 ° C in CuMn alloys makes it possible to design filler metals with brazing temperatures not above those for brass filler metals. However as manganese alloys have high vapor pressure, they segregate at brazing temperatures. Manganese filler metals with Mn content higher than 20% are characterized by low ductility [6].

CHAPTER 3

COPPER-NICKEL ALLOY BRAZING FILLER METALS

The high strength properties and ductility at room and elevated temperatures, good corrosion resistance, low vapor pressure and the fabricality of CuNi alloys have been utilized in designing filler metals for brazing steel. The temperature of brazing with these filler metals is above that of copper brazing. With CuNi base filler metals containing zinc, manganese and phosphorus in such amounts that do not affect the vapor pressure, the brazing temperature can be lowered by the addition of silicon or boron. When added to these alloys, the silicon improves their corrosion and heat resistance and through entering into compounds with nickel the strength upon precipitation hardening [2] .

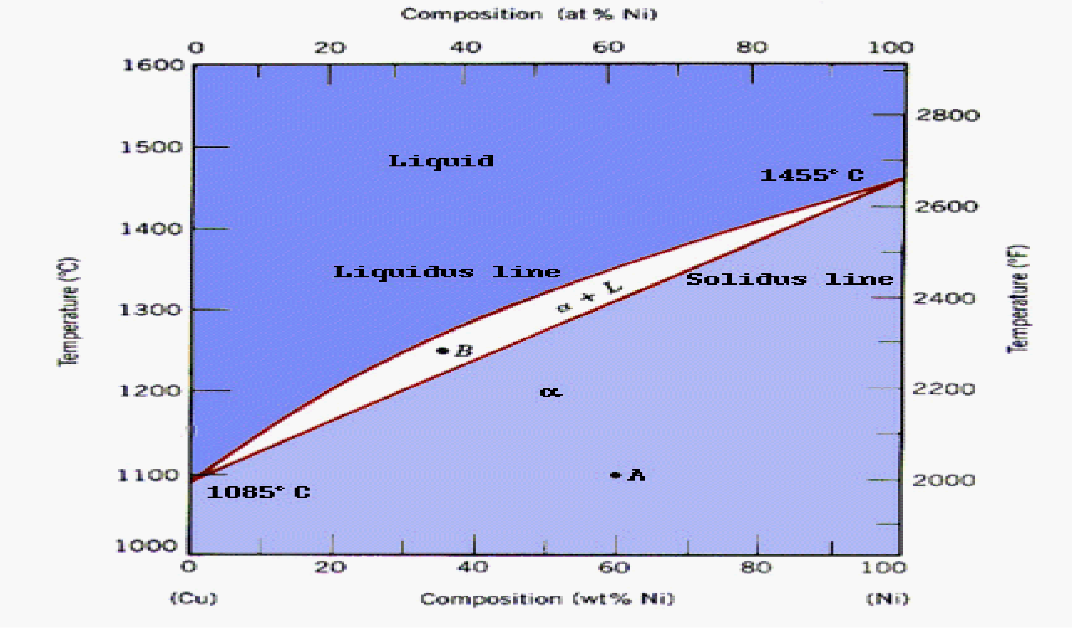

Fig. 3.1 Phase Diagram of Cu-Ni

The addition of nickel or boron to copper filler metals ensures the thermal stability of the resulting brazed joints [2]. Nickel takes copper into a continuous range of solid solutions and raises their melting range Fig 3.1, that is why its percentage in copper alloys is restricted usually to about 2.5 %. The addition of Ni to Cu in a certain amount also reduces the formation of intermetallics [15]

According to Lashko, to strenghten the solid solution and lower the melting point of a filler metal, manganese and zinc can be added along with nickel. Nickel reduces the oxidability of filler metals in the liquid state and improves their wetting ability on the surface corrosion resistant steel. Cu-Mn-Ni alloys are a promising base for the filler metals. Comercial use has been made of self fluxing Cu-Mn-Ni filler metals alloyed with lithium alone or in combination with boron. Two widely used composition of Cu-Mn-Ni filler metals given in [2] are :

1- % 22-26 Mn, % 5-6 Ni, % 0.8-1.2 Fe, % 0.15 – 0.25 Li, balance Cu, with brazing temperature around 1000 ° C in argon atmosphere,
2- % 27-30 Mn, % 28-30 Ni, % 0.8-1.2 Si, % 1-1.5 Fe, % 0.15-0.30 Li, %0.15-0.25 B, balance Cu, with brazing temperature around 1050 ° C.

In case of oxyacetylene heating Cu-Mn-Ni filler metals has tendency to produce porous brazed joints because of the evaporation of manganese into pinholes on overheating.

Copper filler metals are sometimes used in the brazing of high speed cutting tools. It has been suggested by Lashko [2] that the filler metals should be alloyed with nickel (7 to 10 %) and iron (14 to 18 %) to reduce the tendency of medium carbon-high speed steel joints embrittlement. 0.5 to 1.5 % Si and 1.5 to 3 % Mn are added to the filler metal to strengthen the joint and lower the melting point of the filler metal respectively.

CHAPTER 4

NICKEL BASE BRAZING FILLER METALS

Nickel base brazing filler metals provide higher temperature capabilities and improved mechanical properties compared to copper based filler metals.

Pure nickel is sometimes used as a filler metal in brazing titanium, molybdenum and tungsten. The melting point of Ni is 1453 ° C which is much higher than the melting point of most base metals. The melting temperature of nickel is lowered to a required level by alloying them with elements that combine with nickel to form eutectics or lower melting point solid solutions. These are P, In, Si, B, Be, Mn, C, Cr.

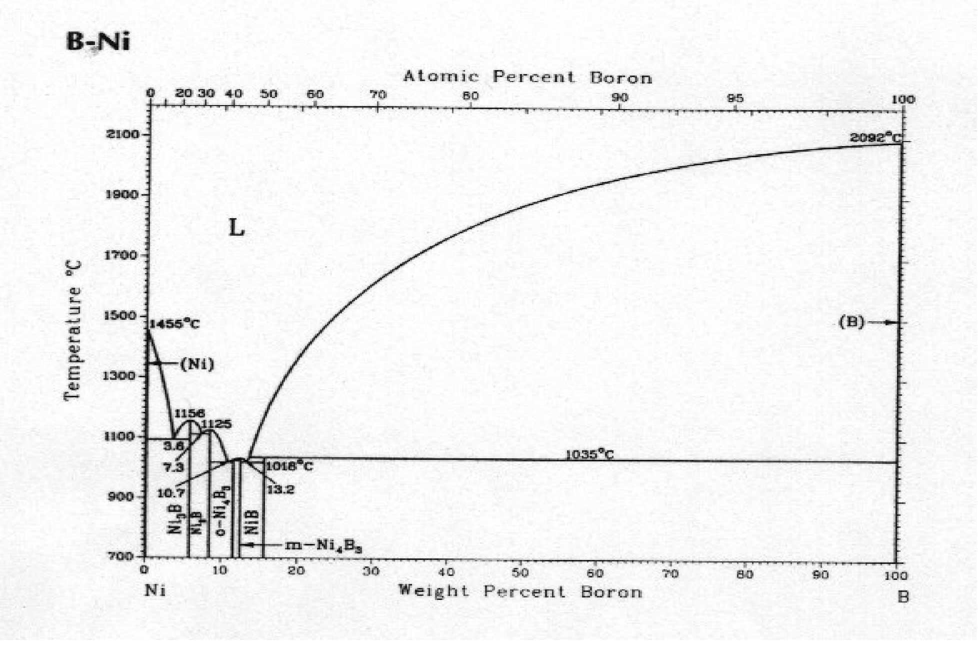

Fig. 4.1 Phase Diagram of B-Ni [9]

When alloyed with nickel, only 3.6 % boron will lower the melting point to 1093 ° C (Fig. 4.1). Boron is only slightly soluble in nickel. The Ni-B eutectic is composed of nickel and brittle constituent Ni_3B. Filler metals containing over 1 % B have lower ductility.

At brazing temperature, boron can be diffused out of the brazed joint and into the adjoining base metal. This diffusion accounts for the drop in filler metal hardness when brazing steel or iron-chromium base metals and the increase in the brazing joint re-melt temperature to above 1370 ° C. An addition of up to 1.75 % cobalt will prevent boron from diffusing into the base metal. [9]

Although boron serves an important function in lowering the melting point of brazing filler metals, when alloyed alone with nickel, boron has some disadvantages. At 2.5 – 3.5 % boron (amounts commonly used in brazing filler metals), the binary nickel-boron alloy will have lower tensile strength and higher hardness, lower corrosion resistance and excessive fluidity when molten [9]. Because of this, all nickel based filler metals containing boron must have additional elements to enhance their physical properties.

Approximately 11.8 % Si alloyed with pure nickel will lower the melting temperature by 330 ° C. NiSi eutectic with about 10 % Si has a melting point of 1125 ° C (Fig 4.2). Due to the interaction between Si and Ni, unlike boron, silicon has slight tendency to penetrate the base metal along the grain boundaries [2].

But when alloyed alone with nickel, silicon like boron will not make a satisfactory brazing filler metal and exhibit low tensile strength, poor ductility and excessive fluidity [9]. A number of filler metals based on NiSi eutectic have been developed. Apart from silicon sometimes these filler metals contain chromium to improve their strength, a specific amount of iron which contributes to their wetting action on the base metal. When 2.75 – 4.5 % silicon and 1.5 – 3.5 % boron are alloyed with nickel, suitable brazing filler metals can be formed. AWS designated BNi-4 [4] brazing filler metal with the composition of 1.9 % B, 3.5 % Si, max 0.06 % C and balance Ni has a wide melting range, free flowing properties, good machinability and low diffusion with most base metals. The melting point of this alloy is 1066 ° C.

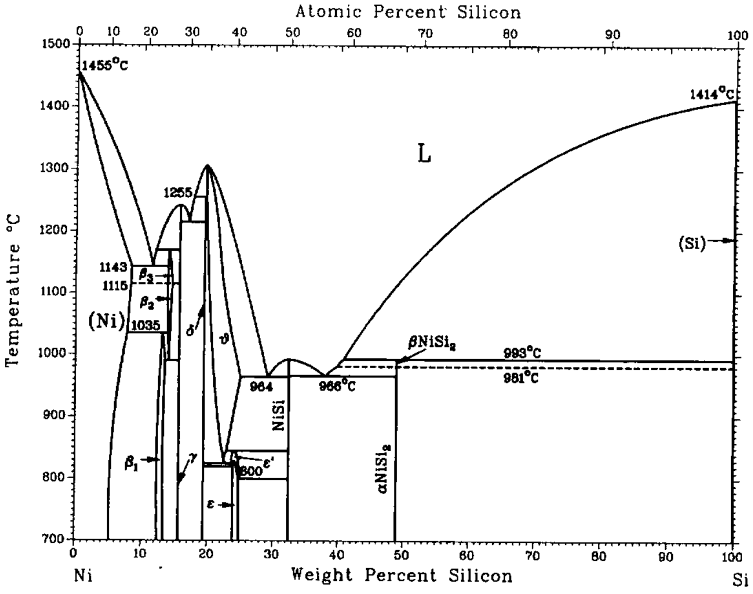

Fig. 4.2 Phase Diagram of Ni-Si [9]

Unlike boron and silicon, phosphorus which is another melting point depressant of nickel, does produce a satisfactory brazing filler metal when alloyed alone with nickel. However it has low strength and ductility and greater fluidity when molten.

In [11] use of NiHf base filler metals in spite of boron, silicon and phosphorus, is suggested. Metalloid elements (boron, silicon and phosphorus) form brittle phases with components of the nickel alloy matrix. There is also a possibility of developing hard phases in the joint during brazing, which could negatively impact mechanical and corrosion properties. the hardness of borides in nickel base filler metals can be as hard as 2000 $HV_{0.05}$. In addition, metalloid elements tend to diffuse into the base metal. In high temperature brazing, this can cause borides to form near the joint. Formation of chromium borides reduces corrosion resistance by depletting chromium from the base metal. The effects of brittle phases can be diminished by a postbreazing heat treatment but this increases the operation costs. The advantages of using hafnium as a melting point depressant in nickel base brazing filler metals are :

1- Melting of NiHf base alloys begins at about 1200 ° C and ends at about 1300 ° C,
2- NiHf eutectic (30 wt% Hf) melts at 1190 ° C ,
3- The Ni-5Hf intermetallic phase at the eutectic structure has a microharness of about 400 $HV_{0.05}$,
4- Excellent oxidation resistance,
5- Melts completely and has good wetting characteristics,

Chromium is soluable in nickel in amounts up to 47 % and improves the strength, heat and corrosion resistance of the brazing filler metal. To maximize the benefits of the chromium addition, the time at brazing temperature can be extended (40-60 minutes) to allow boron to diffuse out of the braze joint and into the base metals being joined. The boron loss will result in a strong, corrosion resistant braze joint with a melting point higher than the original filler metal. 14 wt % addition of chromium to 76 % Ni and 10 % P alloy provides a brazing filler metal with liquidus 888 ° C with increased molten viscosity, higher strength and ductility and higher corrosion resistance. [9]. Boron, or sometimes beryllium, is added to Ni-Si-Cr filler metals to lower their melting temperature and improve the spreadability in dry hydrogen[2]. According to Zhang [13] the formation of Cr_2O_3 at the metal – ceramic joints promote wetting of the filler metal on ceramics, so he suggests addition of 5 % Cr to pure Cu. The source of oxygen for the creation of Cr_2O_3 is assumed to be atmosphere according to the reaction:

$$4Cr + 3O_2 = 2\ Cr_2O_3 \quad [\ 12\ ]$$

Unlike metal – ceramic joints, in the metal – metal joint Cr_2O_3 and oxides of other elements inhibit wetting of the metal surfaces by the filler metals. Because of that oxide removing fluxes and atmospheric controlled furnaces should be used.

By alloying 4.5 % silicon, 3 % boron, 14 % chromium and 4.5 % Fe with nickel a brazing filler metal with a melting temperature of 1038 ° C will be formed. This filler metal is widely used for high strength and heat resistant joints, highly stressed sheet metal structures and other highly stressed components.[9]. Wu [14] found that BNi containing boron and silicon melting point depressants can achieve good wetting and spreading between Inconel X-750 and stainless steel. Wu states that increasing the joining time increases the shear strength of the joint because of the decrease in the intermetallic agglomerate, borides and silicides, along the centerline of the joint.

Manganese is among the most important elements lowering the melting point of nickel and its alloys without an appreciable loss in ductility [2]. The lowest melting point Mn-Ni filler metal is rather ductile in the as cast condition.

CHAPTER 5

EXPERIMENTAL PROCEDURE

5.1 Materials Used

3 different brazing filler metal specimens used in this study are:

Electrolytic Cu
Ni coated Cu sheet
BNi Alloy

Base metals that are brazed in this study are :

Cold Worked Tool Steel
St 37 Steel

As a brazing flux FH 12 (according to DIN EN 1045) is used.

5.1.1 Electrolytic Cu

Specimens of this 99.99 % Cu were supplied, in the form of 150 µm thick sheets. The width of the sheet was 76 mm. The thermal expansion coefficient of electrolytic Cu is 1,8 * 10^{-5} mm / mm ° C.

5.1.2 Ni Coated Cu Sheets

Two interlayers were prepared by coating Cu with Ni. 70 µm thick electrolytic Cu was coated by Ni by leaving it at the electrolytic bath for 20 min. The Ni coating was 17 µm thick, which corresponds to the 24 wt % of the specimen. The second interlayer was prepared by coating 100 µm thick electrolytic Cu by 45 µm thick Ni by leaving it at the electrolytic bath for 40 min.. The width of the coated Cu was 76 mm. The thermal expansion coefficient of 30 wt % Ni - 70 wt % Cu is $1,6 * 10^{-5}$ mm / mm ° C. and that of 10 wt % Ni – 1 % Fe alloy is $1.7 * 10^{-5}$ mm / mm ° C.

5.1.3 BNi Alloy

The alloy was prepared at METU. The composition of the alloy is given in Table 5.3

Table 5.1 Chemical Composition of BNi Alloy

% Ni	% Fe	% Cr	% Si	% B
62	14	16	4	2.5

The coefficient of thermal expansion of the alloy is $1,4 * 10^{-5}$ mm / mm ° C. [2]

5.1.4 Cold Worked Tool Steel

The cold worked tool steel with the chemical composition given in Table 5.4, is an oil-air-vacuum hardening steel which has a good combination of toughness and wear resistance and has a normal hardness in the range of 52 - 58 HRC. The coefficient of thermal expansion at 400 ° C is $1.13 * 10^{-5}$ mm / mm ° C.

Table 5.2 Composition of Cold Worked Tool Steel

% C	% Si	% Mn	% Cr	% Mo	% V
0.5	1.0	0.5	8.0	1.5	0.5

5.1.5 St 37

The composition of St 37 steel is given in Table 5.5. Coefficient of thermal expansion is $0.12 * 10^{-5}$ mm / mm ° C.

Table 5.3 Composition of St 37 Steel

% C	% Mn	% P	% S
0.2	0.9	0.05	0.05

5.1.6 FH 12 Flux

Potassiumhydroxifluorborate, boron and water containing flux has a density of 1.4 gr/cm^3 and a viscosity of 150 to 250 dPaS. The efficient working temperature of the flux is between 550 – 800 ° C.

5.2 Experimental set-up

5.2.1 Sampling

The steels of 70 cm in length are joined to each other as shown in Fig. 5.1.

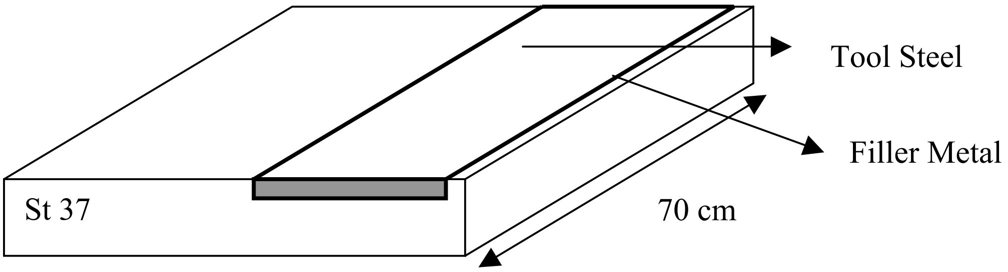

Fig 5.1 Joint Design

These specimens are then cut into 9mm x 9mm x 75mm bars with a notch of 2mm at the tool steel side.

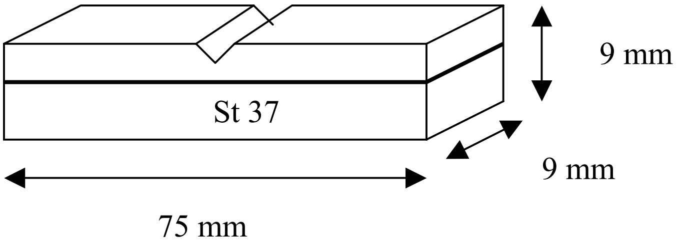

Fig 5.2 Impact Test Sample

Shear test samples were prepared by turning the brazed steels as shown in Fig 5.3.

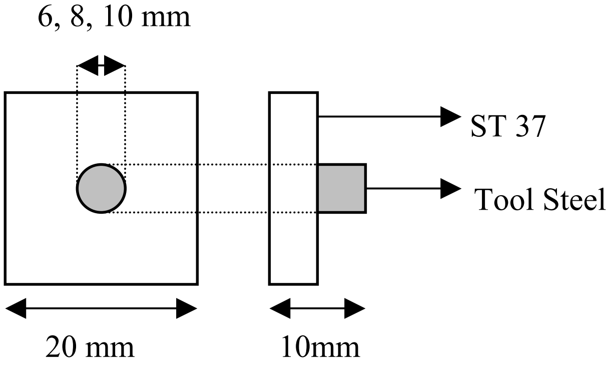

Fig. 5.3 Shear Test Sample

5.2.2 Heat Treatment and Brazing Furnaces

Brazing and austenitization were carried out in a batch furnace. There are two Cr-Ni thermocouples inside the furnace which are 1800 mm apart from each other. There is a max ±7 ° C temperature difference between these thermocouples. The furnace has no preventive atmosphere and fuel oil is used for heating. The maximum operating temperature of the furnace is 1400 ° C.

Heating at the tempering furnace is done by circulation of the hot air. A maximum of 600 ° C can be reached by the tempering furnace and the temperature inside the furnace is measured by Fe-Constans thermocouple.

5.3 Experimental Procedure

After placing the filler metals between the steels, the preparation of the samples for impact testing starts with pre-heating them to 600-650 ° C and holding at this temperature for 20 min. Following pre-heating, the samples are slowly heated to brazing temperature of 1100 ° C or 1180 ° C. The heating rate is 10 ° C / min. The samples are held at the brazing temperature for 30-45 min. depending on the thickness and composition of the brazing filler metal. Then the furnace is cooled down to 1040 ° C which is the austenitization temperature of the tool steel. The TTT graph of the tool steel is shown in Fig 5.5. At 1040 ° C the samples are held for 15-30 min. The effect of austenitizing temperature on hardness, grain size and retained austenite content of the tool steel is as in Fig. 5.6. After austenitization, the samples are left to air cooling. As soon as the temperature of the samples reach 50-60 ° C, they are tempered twice at 540 ° C in order to decrease the retained austenite content. Fig 5.7 shows effect of tempering temperature on retained austenite.

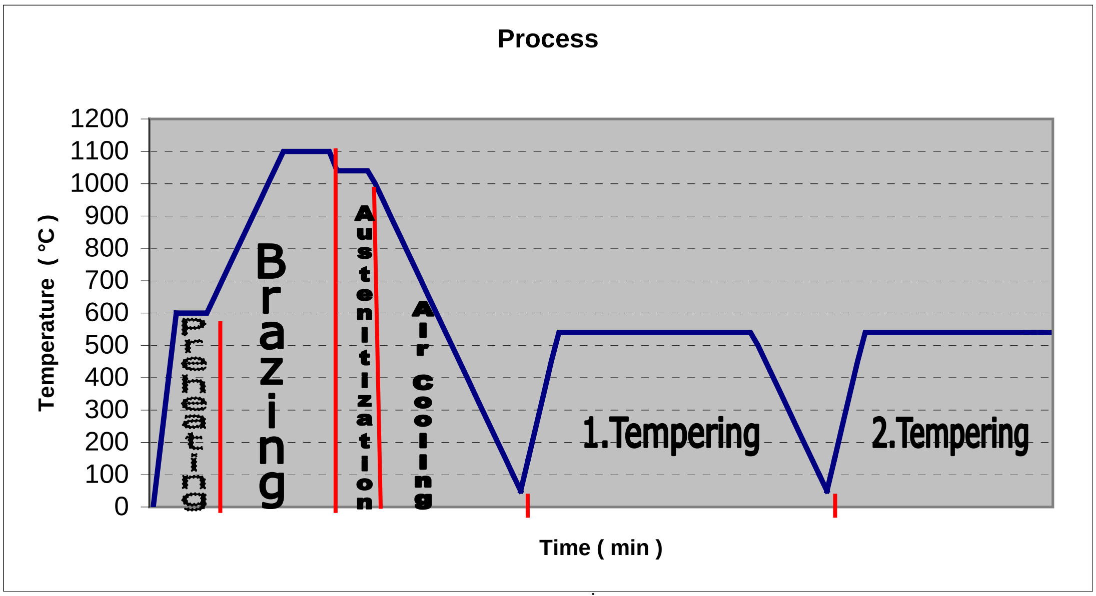

Fig. 5.4 Brazing Process Diagram

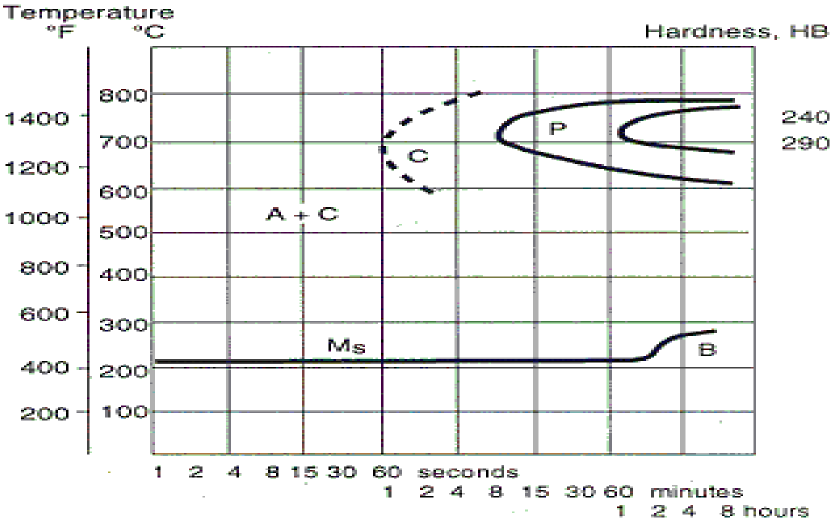

Fig. 5.5 TTT Graph Of Cold Worked Tool Steel [22]

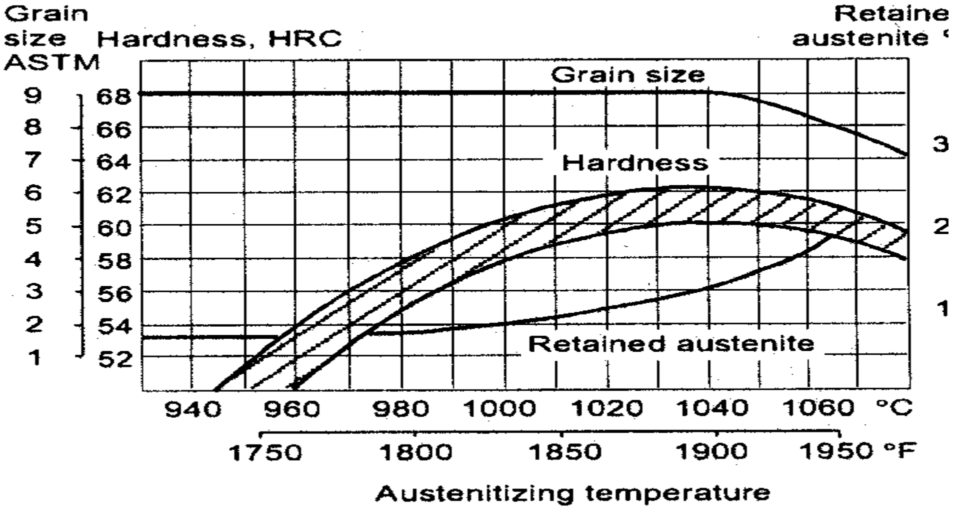

Fig 5.6 Austenization temperature vs hardness, grain size, retained austenite diagram [22]

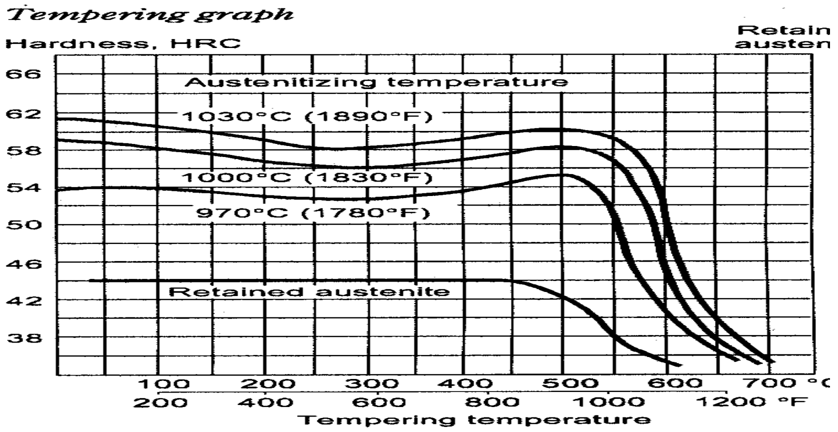

Fig 5.7 Tempering Temperature vs hardness and retained austenite % [22]

5.3.1. Experimental Procedure of Electrolytic Cu Brazing Application

The brazing temperature was 1100 ° C. The sample is held for 30 min. at this temperature to allow the molten Cu to wet the steel surfaces. Beside the brazing temperature and holding time, the remaining of the process is performed as discussed above.

5.3.2 Experimental Procedure of Ni Coated Cu Sheets Brazing Application

According to the Cu-Ni phase diagram, the liquidus temperature of 24 wt % Ni alloy is 1220 ° C. But considering the high diffusion of Ni into Fe it was decided that 1180 ° C would be high enough to melt the brazing filler metal. The holding time at brazing temperature was set to 40 min. 10 wt% Ni coated Cu sheet was brazed at 1200° C and held at that temperature for 40 min. Beside the brazing temperature and holding time, the remaining of the process is performed as discussed above.

5.4 Metallographic Examination

The specimens were metallographically prepared and examined. The primary examination was carried out using an Olympus MG optical microscope, colour photographs were taken by using a Nikon FDX-35 camera that is connected to Nikon-Optiphot-100 type microscope.

Scanning electron microscopy was used as the main examination technique in this study. A JEOL 6400 Scanning Electron Microscope was employed during the study, the spot and line analysis were carried out by a Northern Tracor EDS analysis system attached to the scanning electron microscope.

5.5 X-Ray Diffraction

X-ray measurements were conducted by using a Philips PW 1320 X-ray diffractometer. Co Kα radiation was used as X-ray source.

5.6 Impact Testing

Charphy impact testing of all specimens were carried out by using Tinius Olsen Charpy Impact Test Machiene. The tests were done according to the ASTM Standard Test Methods for Notched Bar Impact Testing of Metallic Materials [20].

5.7 Shear Testing

For better analyzing the bond strength, shear tests were carried out by applying a shear force at the brazed joint. The force applied is in the same direction with the force applied to blade tips under operation. The set up for shear testing is as Fig. 5.8.

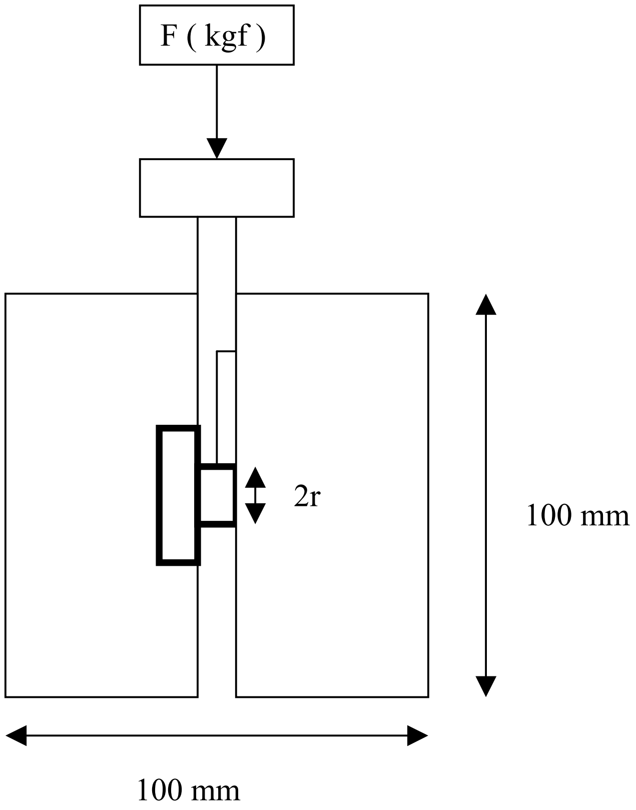

Fig. 5.8 Shear Test Aparatus

The force at the braze tip can be converted to MPa by;

$$\frac{\text{Kgf} * 9.81}{\text{Area}} \qquad \text{Area} = \Pi \, . \, r^2$$

CHAPTER 6

RESULTS & DISCUSSION FOR Cu BRAZING FILLER METAL

Blades manufactured by brazing 8 % Cr containing cold worked tool steel by electrolytic Cu brazing filler metal provides sufficient results in paper cutting. After a certain amount of paper is cut, these blades should be grinded to sharpen the blade tips. Also in higher and continuous impact loads, delamination at the joints was seen. To increase the number of papers cut without a need of a grinding requires other types of tool steels which have higher austenitization temperatures and suitable brazing filler metals for these high austenitization temperatures should be selected.

For better understanding the current situation and to see the improvement by brazing with different filler metals, Cu brazing was used as a reference in this study.

6.1 Mechanical Test Results of Cu Brazing

Table 6.1 Results of Charpy Impact Testing of Cu Brazed Samples

Sample #	Impact Toughness J / cm²	Sample #	Impact Toughness J / cm²	Sample #	Impact Toughness J / cm²
1	16	5	75 (delamination)	9	23
2	17	6	97 (delamination)	10	33
3	97 (delamination)	7	82 (delamination)	11	84 (delamination)
4	42	8	75 (delamination)	12	26

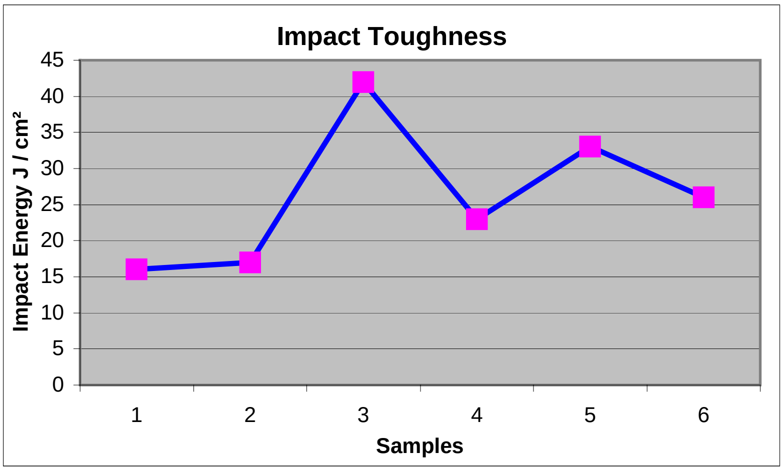

Fig. 6.1 Results of Charpy Impact Testing of Cu Brazing Filler Metal Application. (Delaminated Samples are not considered)

Results show that 50 % of the samples were delaminated when electrolytic Cu was used as the brazing filler metal. The average impact energy of the 6 samples, which were not delaminated, is 26.17 J. Both high delamination occurance and low impact energy values indicate that Cu has relatively poor wetting and bonding capability with the 8 % Cr cold worked tool steel and St 37 steel.

Beside impact tests, shear tests will also tell much about the strength of the bond between the filler metal and base metals.

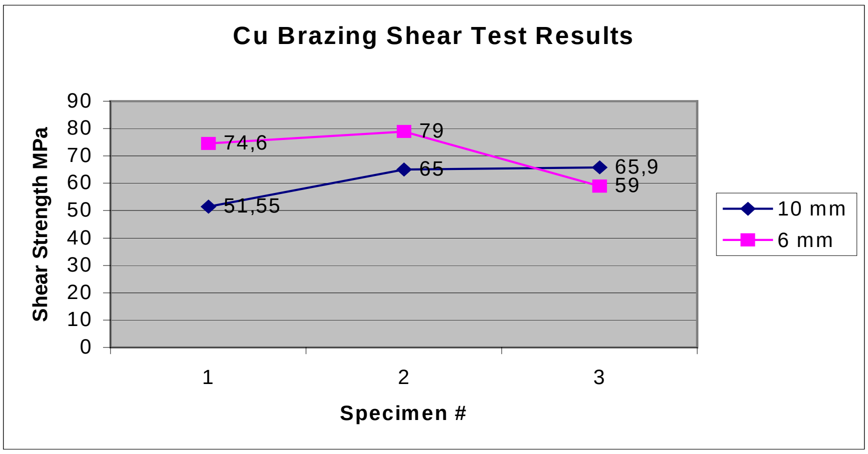

Fig 6.2 Shear Test Results of Cu Brazed Samples

On the joint strength testing, all fractures happened in the St37 / filler metal interface. This indicates that the linkage between tool steel section and the solder are much firmer than that between St37 section and the solder. The average joint shear strength in Cu brazing application is about 65 MPa. Bonding and elemental distribution along the joint were investigated under SEM.

6.2 Metallographic Examination of Cu Brazed Joints

Optical microscope images show limited iron diffusion towards Cu brazing region.

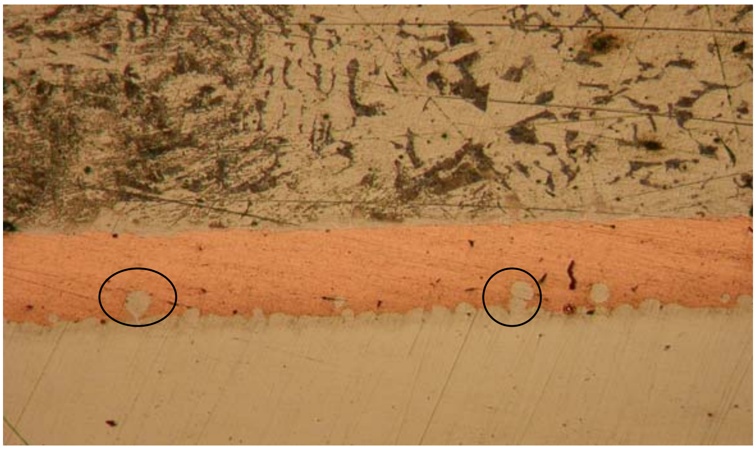

Fig. 6.3 Optical Microscope Image of Iron Diffusion in the Cu Brazed Region in x 200 Magnification

The diffusion coefficient of Cu in Fe at 1050 ° C is given in [3] as 0.557 cm^2 / sec. The atomic diameter of Cu is 2.55 A [9]. This big atomic size and low diffusion rate towards Fe prevents formation of strong bonds between Cu and the steels. The diffusion coefficient of Fe in Cu at 1070 ° C is 1.334 cm^2 / sec and atomic diameter of Fe is 2.52 A. According to S.Collard Churchill [6] brazing copper takes up 2.3 % iron at 1130 ° C in 30 minutes and 1.6 % iron in 2 minutes. The elemental distribution in brazed region is analyzed by line scan and x-ray mapping techniques. The scanned line was indicated in SEM image in Fig. 6.4.

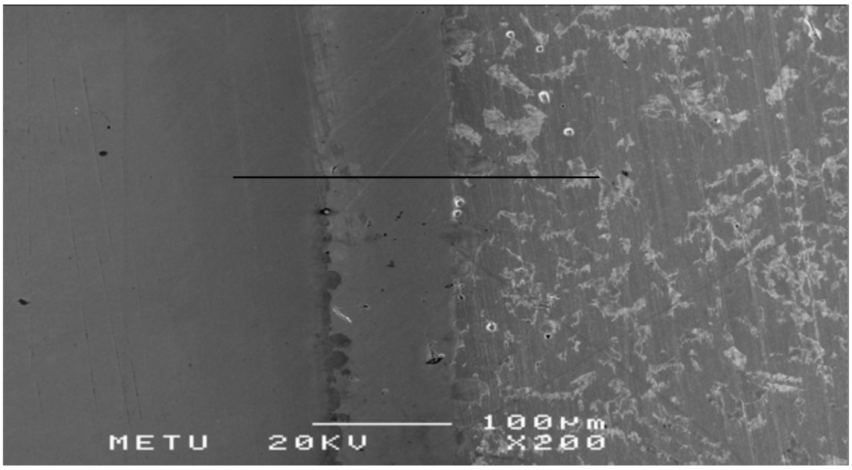

Fig. 6.4 SEM Image of Cu Brazed Region

As seen in figures, there is a limited diffusion of Fe towards the brazed region. Thus the bond is mainly formed by diffusion of iron in molten copper.

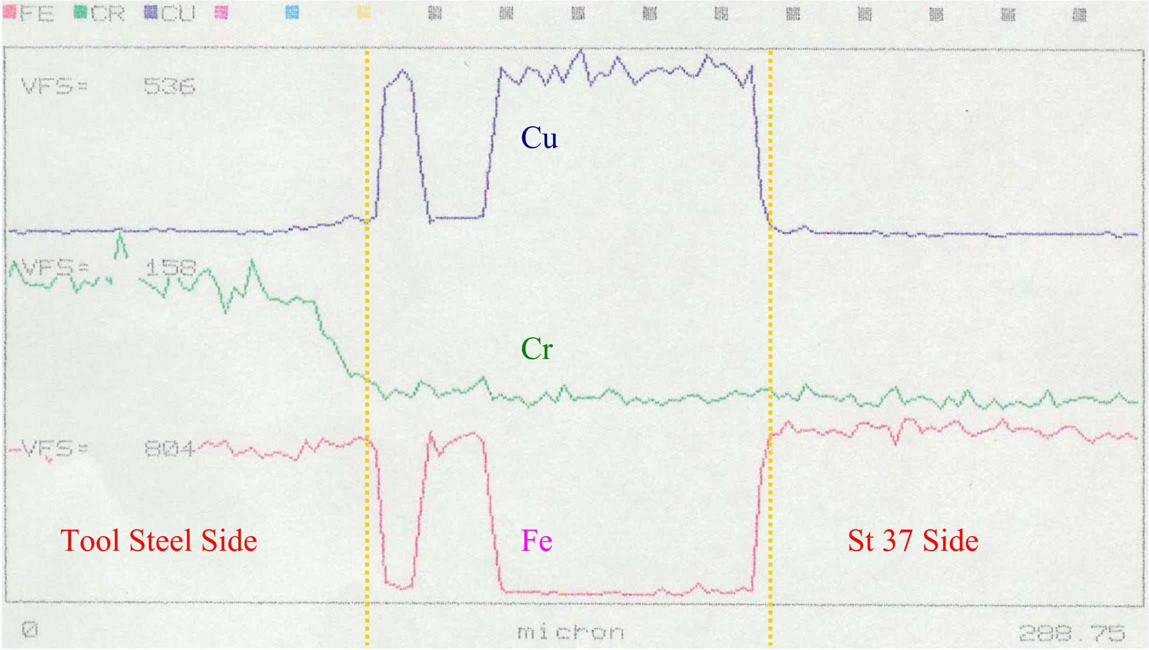

Fig. 6.5 Line Scan of Cu Brazed Joint

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Fig. 6.6 X-Ray Mapping of Cu Brazed Joint

Optical microscope, x-ray mapping and line scan images indicate that Fe in brazed region had diffused from the tool steel side of the joint. The separation of the joint from the St 37 / filler metal interface under shear loading is because of the relatively sound bond between the tool steel and filler metal caused by diffusion of Fe from the tool steel side. Just close to the brazing interface of the tool steel Cr decreases and no Cr is seen in the brazed region.

The thermal expansion coefficient of Cu is $1.8 * 10^{-5}$ mm / mm ° C. The slight difference between the thermal expansion coefficients of Cu and tool steel will not cause a serious stress as the length of the specimen is 70 cm.

Alloying of Cu with other metals such as Zn, B, P, Mn, Si, generally depresses the melting point. This will cause loss of the brazing metal during austenitization of the tool steel which will result with a weak joint. On the other hand, Ni addition to Cu not only increases the melting point but also increases the strength of the joint. Because of that Ni alloying with Cu was considered in this study.

CHAPTER 7

RESULTS & DISCUSSION FOR Cu – Ni BRAZING FILLER METAL

From the fact that nickel reduces the oxidability of filler metals in the liquid state and improves their wetting ability on the surface corrosion resistant steel, it was believed to achieve stronger bonding between the Cu-Ni brazing filler metal and the steels. Also the addition of copper in nickel reduces the formation of intermetallics.

Two interlayer were prepared by coating electrolytic Cu with Ni. The first interlayer has a Ni content of 24 wt % and prepared by leaving 70 µm electrolytic Cu sheet in Ni bath for 20 minutes. The final thickness of the sheet was 87 µm. The second interlayer has a Ni content of 10 wt % and prepared by leaving 100 µm electrolytic Cu sheet in Ni bath for 40 minutes. The final thickness of the sheet was 145 µm.

7.1 Mechanical Test Results of Cu-Ni Brazing

Table 7.1 Results of Impact Testing of 24 wt% Ni Coated Cu Brazed Samples

Sample Number	Impact Toughness J / cm²	Sample Number	Impact Toughness J / cm²	Sample Number	Impact Toughness J / cm²
1	45	7	123 (Delamination)	13	126 (Delamination)
2	150 (Delamination)	8	130 (Delamination)	14	26
3	30	9	32	15	24
4	24	10	124 (Delamination)	16	33
5	38	11	44	17	26
6	32	12	134 (Delamination)	18	36

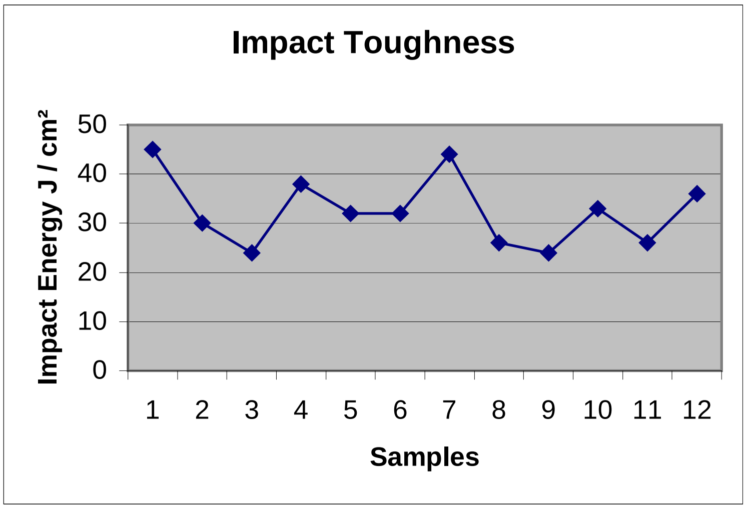


Fig. 7.1 Results of Charpy Impact Testing of 24 wt% Ni Coated Cu Sheet Brazing Filler Metal Application (Delaminated Samples are not considered)

As seen in Fig. 7.1 and Table 7.1, results indicate a considerable decrease in the occurance of delamination and increase in the average impact energy. Only 33 % of the samples were delaminated under impact loading and the average impact energy of samples, which were not delaminated, is 32.5 J. The results indicate that the Ni addition increases the joint strength by forming stronger bonds with the base metals.

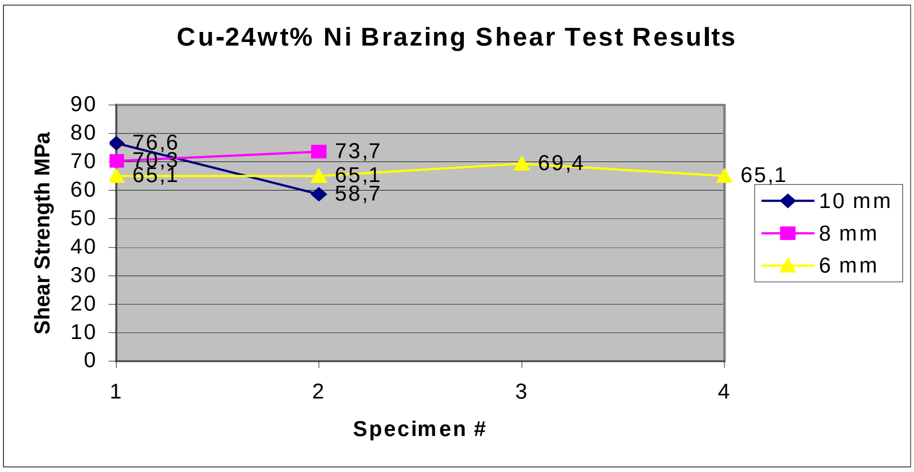


Fig 7.2 Shear Test Results of Cu-24 wt % Ni Brazed Samples

Beside impact test results, joint shear test results also indicate an improvement with an average shear strength of 68 MPa. The effect of Ni addition to filler metal is investigated under optical and scanning electron microscopes.

Table 7.2 Results of Charpy Impact Testing of 10 wt % Ni Coated Cu Brazing Filler Metal Application

Sample Number	**Impact Toughness J / cm²**	**Sample Number**	**Impact Toughness J / cm²**
1	176 (Delamination)	6	168 (Delamination)
2	199 (Delamination)	7	192 (Delamination)
3	186 (Delamination)	8	182 (Delamination)
4	200 (Delamination)	9	194 (Delamination)
5	184 (Delamination)	10	192 (Delamination)

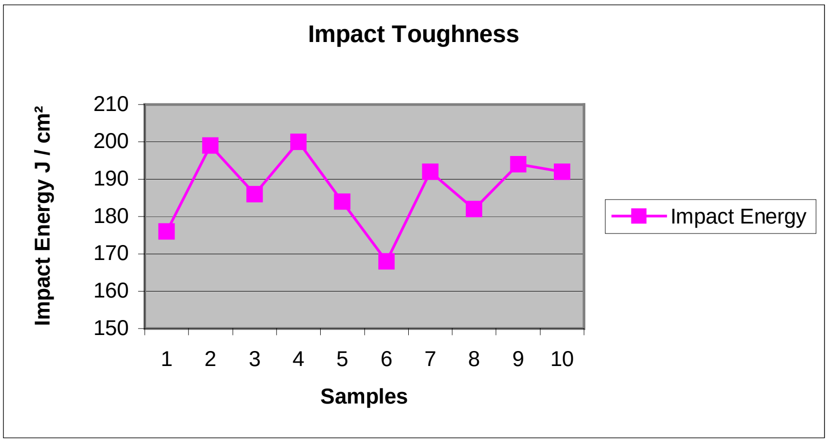


Fig. 7.3 Results of Charpy Impact Testing of 10 wt% Ni Coated Cu Sheet Brazing Filler Metal Application

Decreasing the Ni content to 10 wt % caused 100 % delamination. By examining the fracture surfaces it was seen that no fracture had occurred in St37 section. The ductility of this section in this application is believed to occur because of the heat treatment cycle, in which the preheating was different from that of the 24 wt % Ni brazing application. In 24 wt % Ni brazing the temperature in the furnace was 600 °C, on the contrary the furnace temperature in 10 wt % Ni brazing was 25 °C and it was gradually increased to preheating temperature.

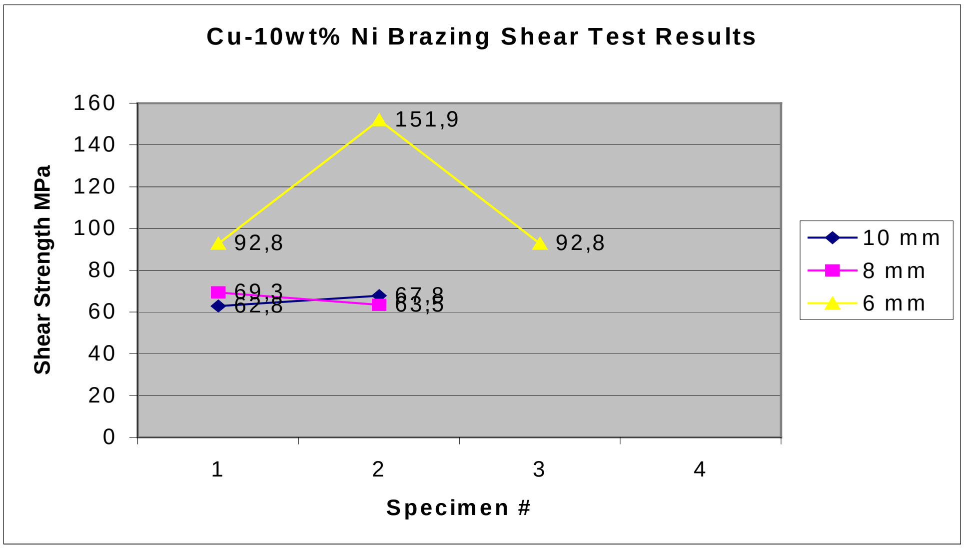


Fig 7.4 Shear Test Results of Cu-10 wt % Ni Brazed Samples

Shear test results of 10 wt % Ni brazed joints show a slight improvement in joint shear strength with an average of 65.8 MPa. In this calculation 6 mm joints were not calculated as there seems to be an experimental error due to the preparation of the specimens. The difference in microscope images of 24 wt % Ni and 10 wt % Ni brazed joints will show the importance of Ni in filler metals.

7.2 Metallographic Examination of Cu-Ni Brazed Joints

The liquidus temperature of 24 wt% Ni – Cu alloy is 1220 ° C, so it may be thought that the brazing temperature of 1180 ° C will not be high enough to form a full molten filler metal in the joint. However, as nickel is a very active metal and has high diffusivity in iron (9.4 cm^2 / sec at 900 ° C [5]) and also in copper (1.89 cm^2 / sec at 900 ° C [5]) transition liquid phase like bonding was seen. During pre-heating and heating to the brazing temperature, the fast diffusion of nickel towards base metals leaves the interlayer with an actual Ni composition lower than 24 wt%. At 1085 ° C Cu melts and with continuing solute diffusion, the melt composition becomes nickel and iron enriched. When heating the assembly to brazing temperature the diffusion of iron in nickel-copper interlayer increases [7], resulting an increase in the melting temperature and eventual isothermal solidification.

Additional time at brazing temperature permits solid state diffusion to dilute the solute concentration even further, resulting in improved mechanical properties of the joint [17]. Thus the kinetics of a transient liquis phase bonding depend on the diffusivity of solute (from the liquid phase) in the solid parent material [19].

Optical microscope images and line scan figures indicate that by increasing the Ni content activated iron amount increases so higher Fe diffusion towards joint area is seen.

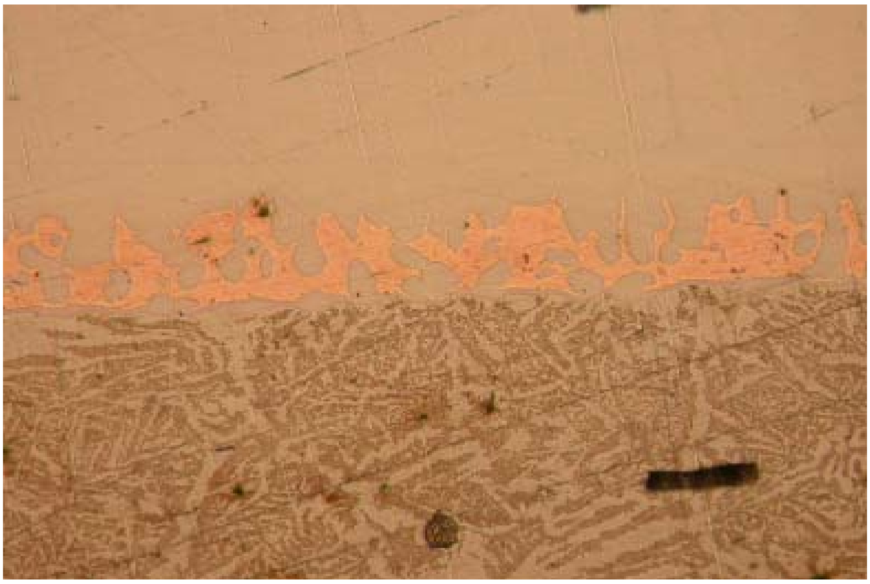


Fig. 7.5 x200 Optical Microscope Image of Cu-24 wt % Ni Brazed Region

The diffusion coefficient of iron in nickel at 1050 ° C is 0.78 cm^2 / sec and at 1200 ° C it increases to 0.98 cm^2 / sec. Also according to Smiryagin and Kvurt [7] the presence of copper in nickel increases the diffusion of iron in nickel so dissolution of iron is seen. As copper has very low diffusivity both in nickel (0.56 cm^2 / sec at 1150 ° C [5]) and in iron (0.56 cm^2 / sec at 1150 ° C [5]) copper can easily be seen by looking at the fracture surface.

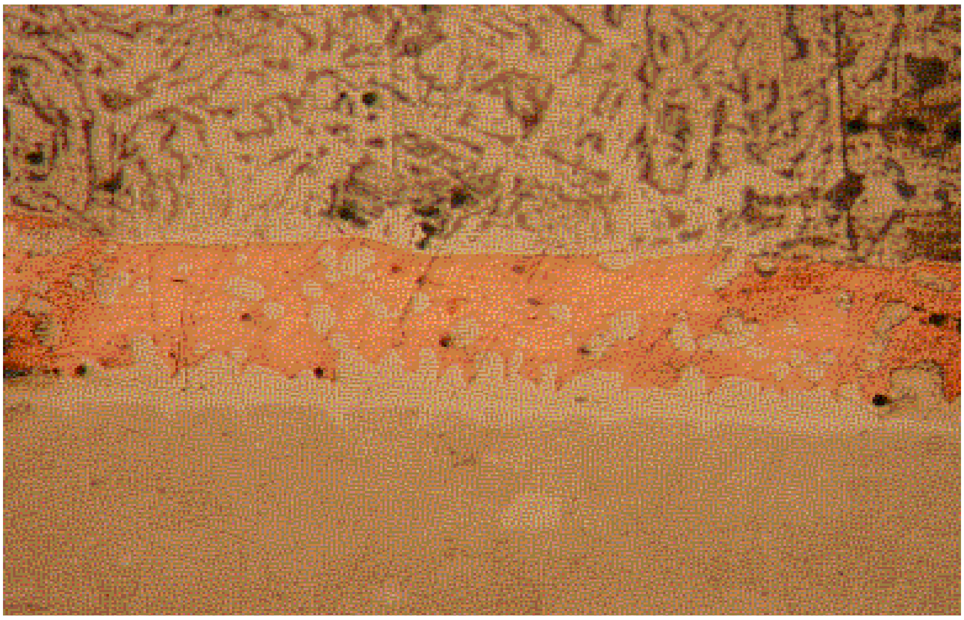


Fig. 7.6 x200 Optical Microscope Image of Cu-10 wt % Ni Brazed Region

Just by considering the optical microscope images of Cu-Ni brazed joints it is clearly seen that Fe diffusion towards joint area is higher in 24 wt % Ni brazing filler application. It will also clearly seen in line scan and x-ray mapping figures that the Fe at the brazed region is mostly diffused from the tool steel side of the joint which owns to the stronger bond at this side of the joint.

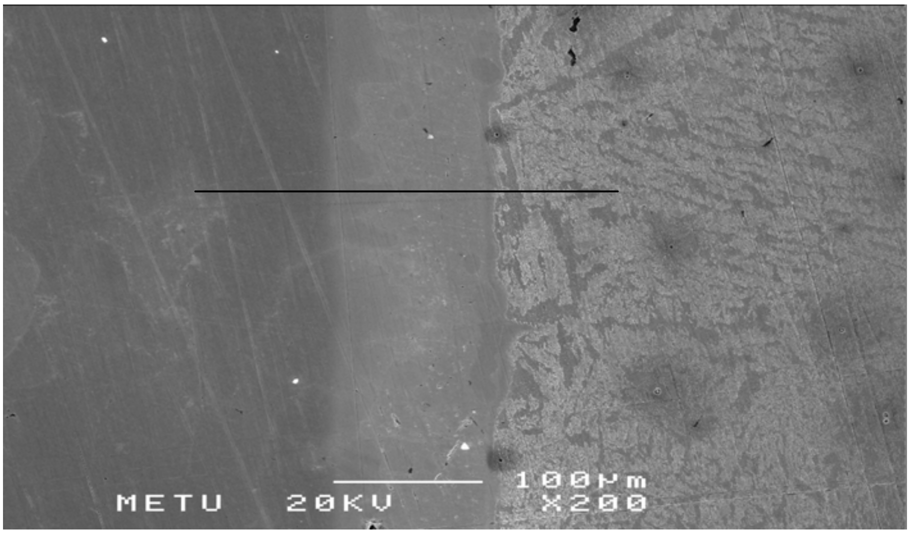


Fig. 7.7 SEM Image of Cu-24 wt% Ni Brazed Region

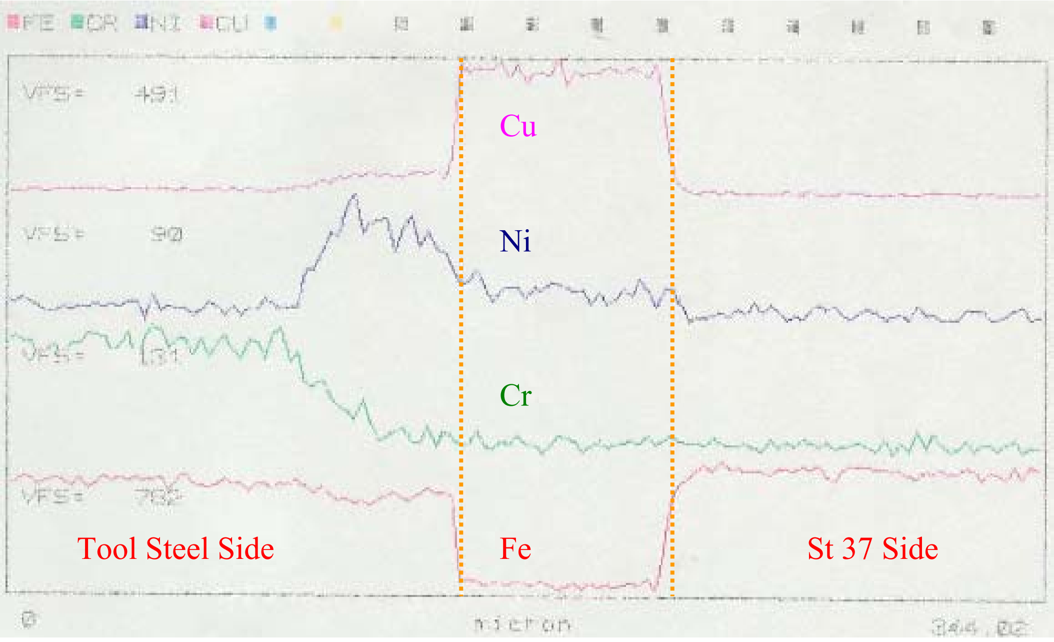
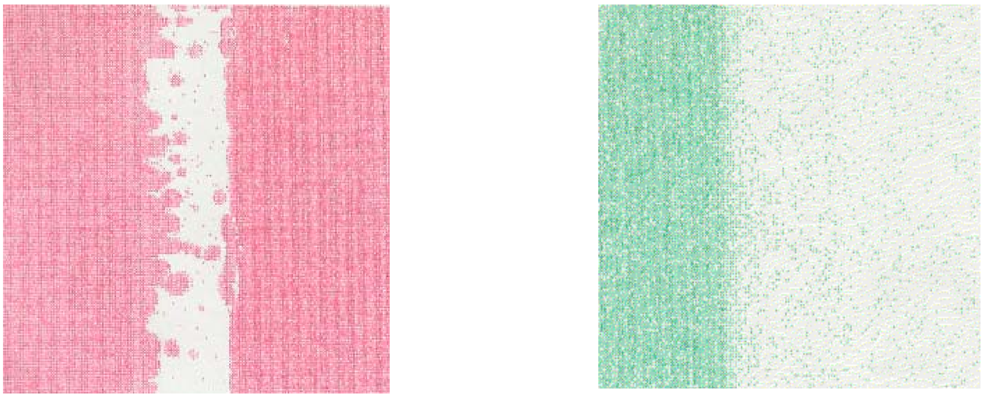


Fig. 7.8 Line Scan of Cu-24 wt% Ni Brazed Joint



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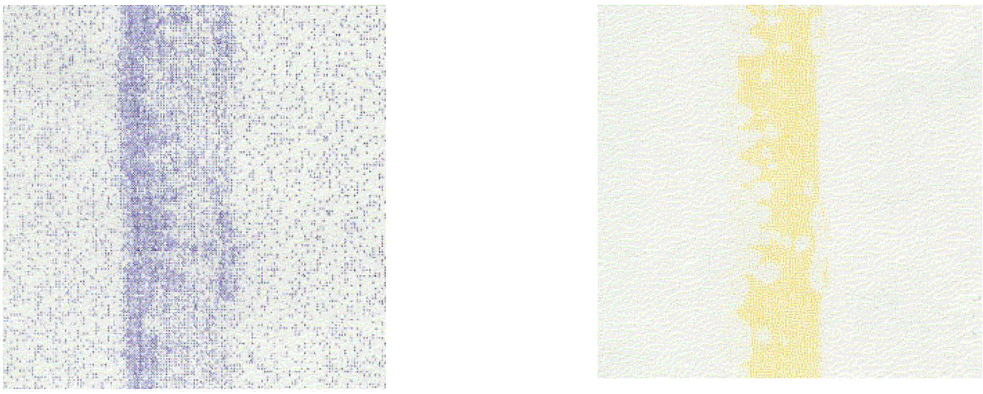

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Fig. 7.9 X-Ray Mapping of Cu-24 wt % Ni Brazed Joint

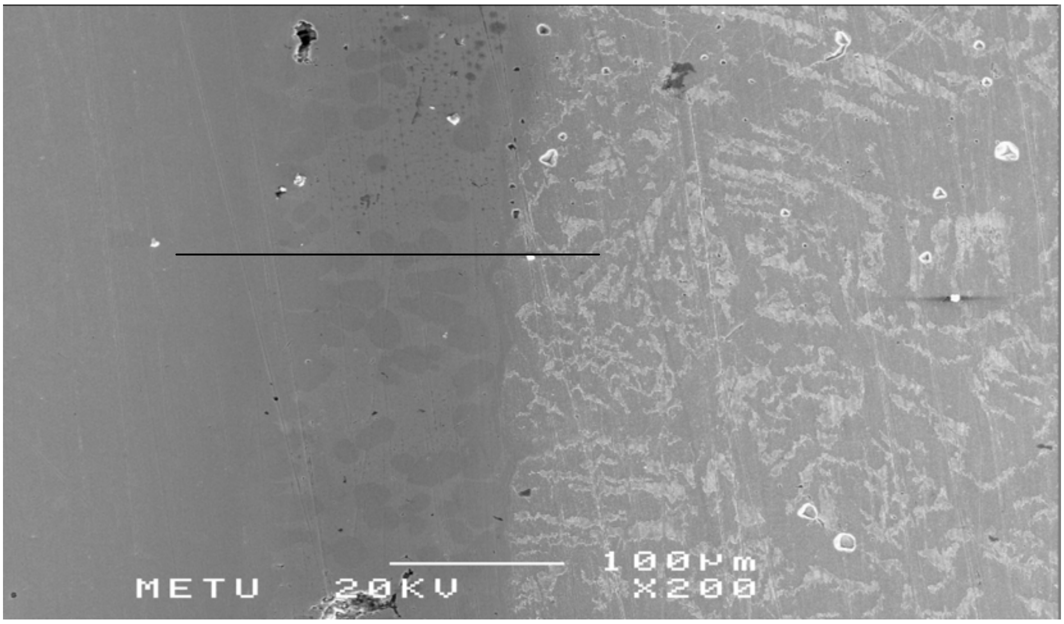


Fig. 7.10 SEM Image of Cu-10 wt% Ni Brazed Region

Fig. 7.11 Line Scan of Cu-10 wt% Ni Brazed Joint (Tool Steel Side)

Ni atoms diffuses interstitially in sites of Cr atoms. The decrease in Cr content near the brazing interface is believed to occur due to oxidizing of Cr atoms.

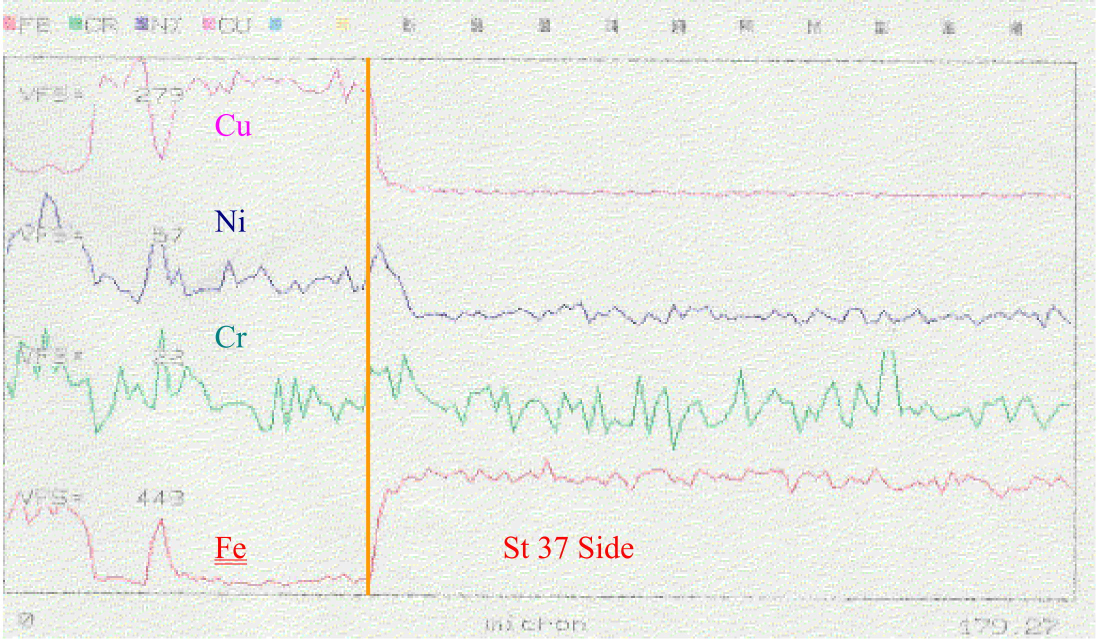
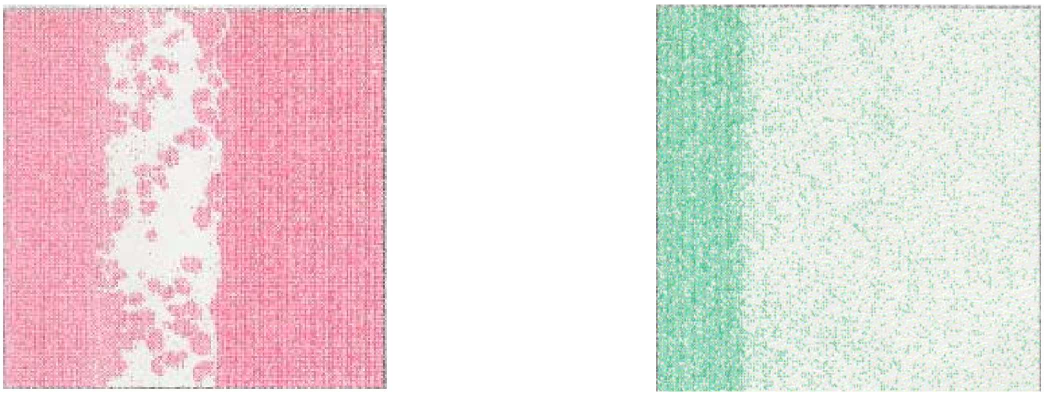


Fig. 7.12 Line Scan of Cu-10 wt% Ni Brazed Joint (St37 Side)



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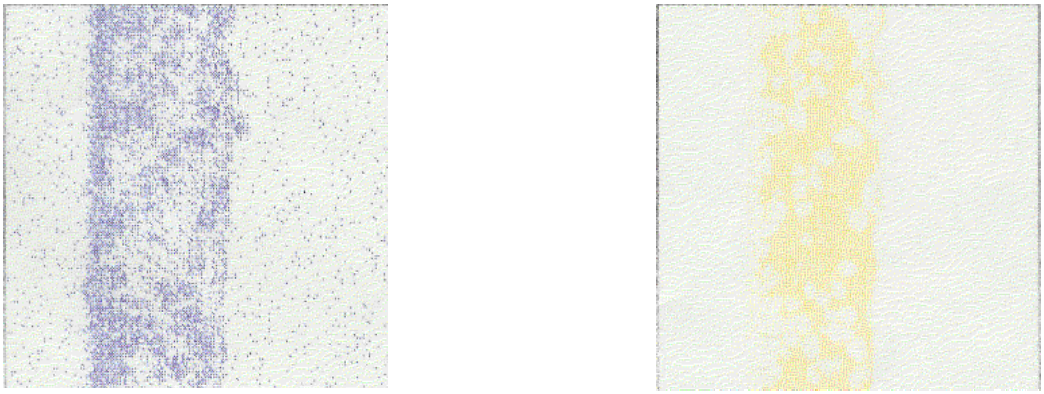

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Fig. 7.13 X-Ray Mapping of Cu-10 wt % Ni Brazed Joint

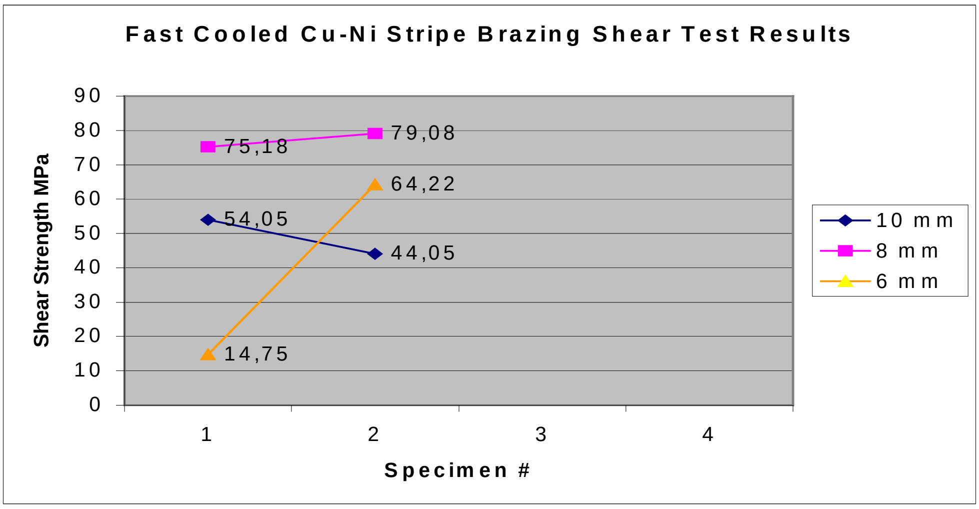


Fig. 7.14 Shear Test Results of Fast Cooled Cu-Ni Stripe Brazed Samples

According to S.R. Cain [16] the time required to complete the bond increases with increasing layer thickness owing to the larger amount of liquid to be solidified. For this reason 10 wt % Ni coated Cu filler metal was brazed at 1200 ° C for 40 min. This temperature may not be sufficient to melt the filler metal. But to prevent the grain growth of base metals, to maintain the mechanical properties of the tool steel, and for economical reasons the brazing temperature and the holding time can not be increased further.

Although the increase in Ni content both improved the shear strength and impact toughness values, due to high melting point of Ni further increase in Ni content is not possible.

The thermal expansion coefficient of Ni is $1.7 * 10^{-5}$ mm / mm ° C which is very close to that of Copper, so Ni addition will not create additional stress due to thermal expansion coefficient difference. On the contrary, the ductile Cu-Ni filler metal will limit stresses arising between the base metals with different thermal expansion coefficients.

CHAPTER 8

RESULTS & DISCUSSION FOR Ni BASE BRAZING FILLER METAL

In the previous work it was seen that Ni has a higher chemical affinity with the base metals, so stronger joints were obtained. This result directed the study to search for a filler metal which is based on Ni. As the melting point of Ni is so high, there should be some melting point depressant elements and also some higher strength elemets in the alloy.

62 % Ni, 14 % Fe, 16 % Cr, 4 % Si, 2.5 % B alloy was prepared as a filler metal. The anticipated alloy was BNi-1 alloy with the chemical composition 74 % Ni, 4.5 % Fe, 14 % Cr, 4.5 % Si, 3 % B which is widely used for high strength and heat resistant joints and in highly stressed sheet metal structures and other highly stressed components.

As seen, there is a big difference in Ni and Fe compositions of the actual alloy and the anticipated alloy. This was due to the lack of finding pure B to prepare the filler metal alloy. Because of that, ferro-boron with 15 % boron is used as a raw material to prepare the filler metal alloy.

The main disadvantage of high Fe content in the alloy is the carbon coming from the ferro-boron. During preparation of the alloy there was a risk of combination of carbon with chromium to form brittle chromium carbide intermetallics. The x-ray and scanning electron microscope analysis support this phenomena. The intermetallics can easly be seen under optical microscope, Fig. 8.1.

After the X-ray and scanning electron microscope analysis of the alloy, it was found that the intermetallics are chromium nickel silicon carbides. Because of these intermetallics, this alloy is not used as a brazing filler metal. Boron addition to the filler metals may be done by applying borax to the surfaces of the filler metals.

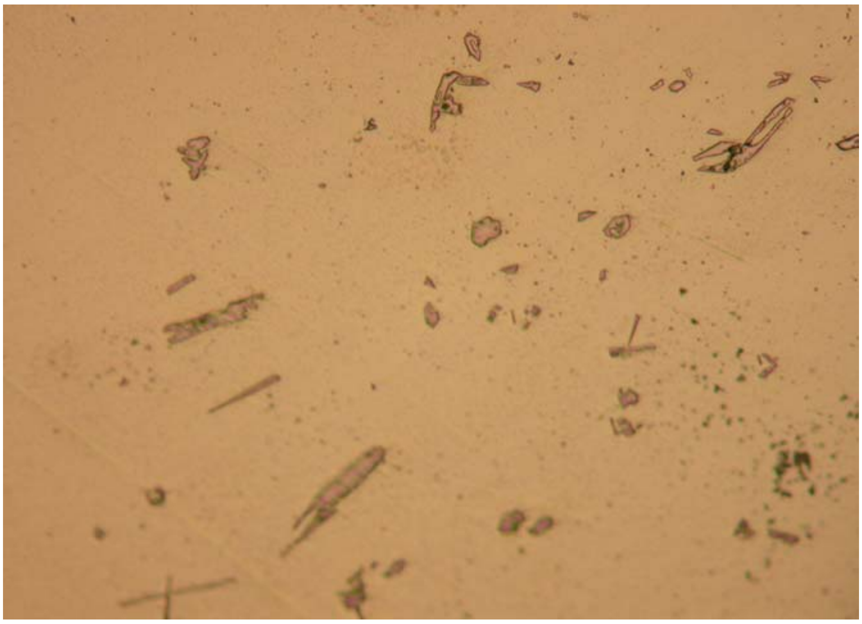


Fig 8.1 Intermetallics under optical microscope in x200 magnification

It was known that high Fe content in the filler metal will cause formation of an independent phase detrimental to corrosion resistance. In addition, metalloid elements tend to diffuse into the base metal. In high temperature brazing, this can cause borides to form near the joint. Formation of chromium borides reduces corrosion resistance by depletting chromium from the base metal. As paper cutting blades are subjected to continuous corrosive environment during their usage, there would be a corrosion defect at the interface in case high Fe containing BNi alloy had been used.

CHAPTER 9

CONCLUSIONS

From this study the following conclusions were drawn:

1- Joint delamination was observed in each of the brazing filler metals used in this study.

2- Half of the joints, brazed by Cu filler metal, delaminated under impact loading. The high delamination occurance and low impact energy values were due to the limited interdiffusion between Cu filler metal and the base metals.

3- By coating the Cu sheet with Ni, delamination occurance decreased and impact energy increased.

4- The combination of carbon and chromium formed brittle chromium carbide intermetallic in the filler metal. Because of that BNi alloy was not used as a brazing filler metal.

5- Transient Liquid Phase Bonding, by high diffusion of Ni into tool steel and into core Cu sheet, owns to the high strength joints.

6- 24 wt % Ni coated Cu brazing filler metal is superior than the other alternatives in both strength and material consumption.

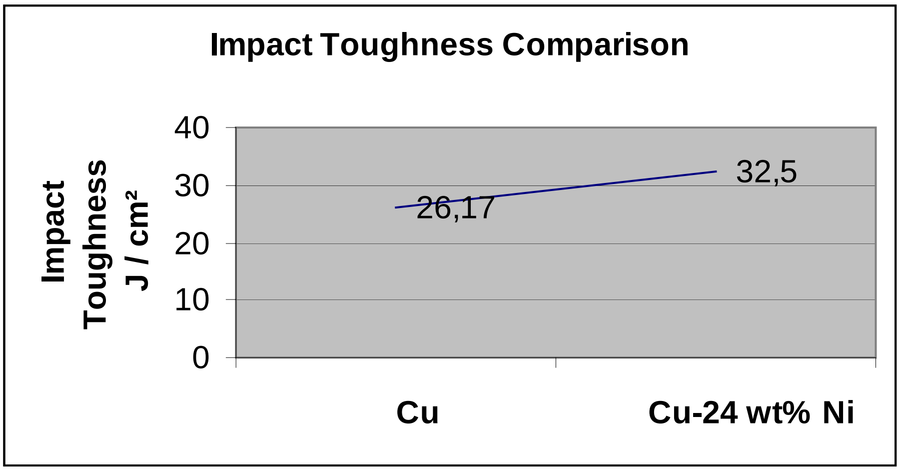
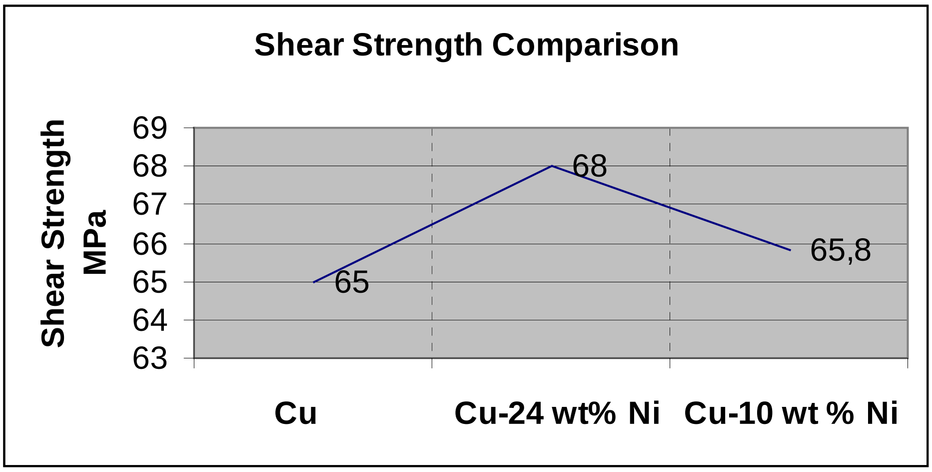


Fig. 9.1 Impact Toughness Comparison of The Filler Metal Applications



9.2 Shear Strength Comparison of the Filler Metal Applications

7- The linkage between tool steel section and the solder are much firmer than that between St 37 section and the solder .

8- Fe is the main element in forming bonds. Ni activates the Fe atoms and causes them to diffuse towards the brazed region.

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